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DETERMINATION OF ALUMINUM CONCENTRATION IN SEAWATER BY COLORIME--ETC(U)
NOV 72 C A GREENE, E N JONES

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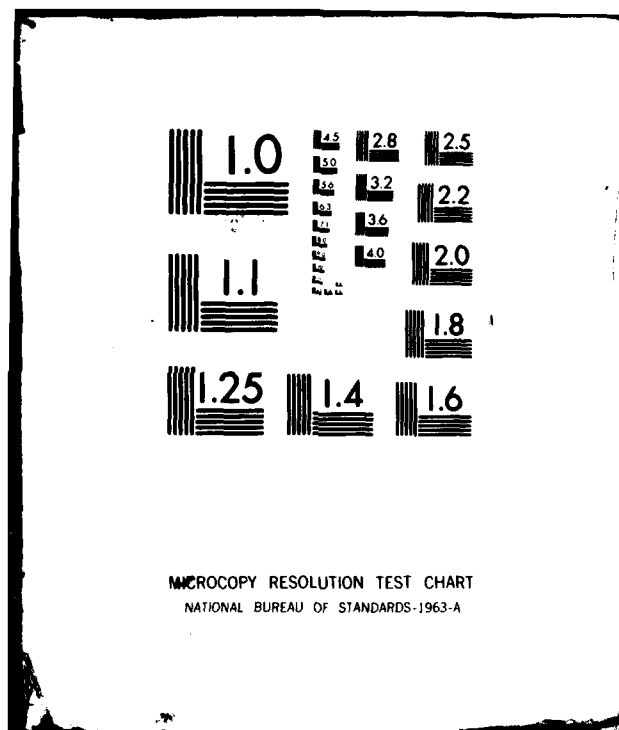
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(6) DETERMINATION OF ALUMINUM CONCENTRATION
IN SEAWATER BY COLORIMETRY AND
ATOMIC ABSORPTION SPECTROSCOPY.

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NAVAL UNDERWATER SYSTEMS CENTER
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ABSTRACT

Aluminum, although present in very low concentrations in seawater, may be responsible for the excess sound attenuation observed in seawater below 10 kHz. However, concentration values cited in the literature vary widely (10^{-3} to 0.5 ppm), so it has become necessary to redetermine the aluminum concentration of seawater. Accordingly, both colorimetric and atomic absorption analyses were performed on 6 recent samples of known depth from a location off Bermuda; 8 older surface water samples from different locations were analyzed by the atomic absorption method.

Results showed the aluminum concentration to increase gradually with depth. Concentration values obtained by the colorimetric method ranged from 0.2 ppm at 100 m to 0.4 ppm at 2500 m, while the atomic absorption method yielded values ranging from .089 ppm at 100 m to .122 ppm at 2500 m. Analysis of the 4 year old surface water samples showed that the aluminum concentrations were negligibly small. Both methods of analysis appear to be well suited for the determination of aluminum in seawater though improved accuracy would be possible by increasing the volume of the water samples.

ADMINISTRATIVE INFORMATION

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INTRODUCTION

Aluminum is present in very low concentrations in the oceans. It can exist both in solution and as particulate, although its source is almost exclusively terrigenous particulate matter, such as silts and clays. The low concentration of aluminum in seawater is primarily due to the high reactivity of soluble aluminum species with the marine environment. Once introduced into the ocean rapid removal occurs transforming soluble aluminum into insoluble materials which sink to the bottom as sediment. As a result, aluminum has a relatively short resident time in the ocean (on the order of 199 years.¹) This means that it is highly probable that localized variations in the concentration of aluminum do occur owing to the lack of time required to achieve homogeneity, since most of the aluminum in particulate form settle out locally before complete mixing throughout the oceans can occur.

Although efforts to determine the concentration of aluminum in seawater appear to be few in number, there does seem to be a wide range in the values reported in available literature. Goldberg¹ simply lists a flat value of 0.01 ppm for aluminum, with no reference as to the analytical method used. Haendler and Thompson², utilizing a colorimetric technique for determining the aluminum concentration, reported average monthly values which varied seasonally from 0.216 ppm to 0.432 ppm for near-shore surface samples collected over a one year period, and values ranging from 0.567 ppm at the surface to 1.754 ppm at 2000 meters for samples collected at a location on the continental shelf in the northeast Pacific. Sackett and Arrhenius³ report a dissolved aluminum concentration in coastal California waters and the Weddell Sea averaging 1 microgram per liter (ppb). Since reported values differ by as much as 3 orders of magnitude, as these do, it is difficult to accept any as the correct aluminum concentration.

As part of the investigation into the origin of the excess sound attenuation anomaly observed in seawater below 10 kHz, chemical relaxation processes are being considered. Pressure-jump experiments⁴ have revealed the $\text{Al}_2(\text{SO}_4)_3$ solutions relax with about the same relaxation time (160 microseconds) as is indicated by acoustic data

TM No.
TA131-285-72

from sound measurements in the ocean. However, the magnitude of the observed absorption appears to be too large to be accounted for by the low concentration of aluminum reported in seawater. To completely evaluate the importance of the aluminum relaxation in seawater it is necessary to resolve the discrepancy of aluminum concentration in the sea.

Therefore, we have attempted to redetermine the concentration of aluminum in seawater by performing both atomic absorption and colorimetric analyses on the same water samples and comparing the results. Both determinations were performed on 6 seawater samples of known depth (ranging from 100 m to 2500 m) obtained from Ocean Acre 14 off Bermuda in June, 1972 and stored in 1-liter polyethylene bottles. The atomic absorption analysis was also repeated on 8 surface samples obtained from different locations in the western Atlantic and eastern Pacific Oceans during the summer of 1968 and stored under refrigeration in polyethylene bottles.

A. COLORIMETRIC DETERMINATION

The colorimetric determination was performed using basically the same procedure as Haendler and Thompson.² 8-hydroxyquinoline precipitates aluminum as $\text{Al}(\text{C}_9\text{H}_7\text{ON})_3$ from solutions with a pH between 4.2 - 9.8. The precipitate is filtered and redissolved in ethanol-HCl solution and coupled with diazotised sulfanilic acid. The reddish-orange color which develops upon addition of NaOH is stable for several hours and easily comparable to a blank.

TM No.
TA131-285-72

REAGENTS

8 - hydroxyquinoline solution: 1 g. of 8 - hydroxyquinoline is mixed with 1 ml of glacial acetic acid and added to 100 ml boiling water. The solution is kept at boiling temperature with frequent stirring until the reagent is dissolved. The solution is then cooled and filtered.

Sodium acetate solution: A saturated solution is prepared at 50°C and permitted to cool. The solution is then kept in contact with excess solid sodium acetate.

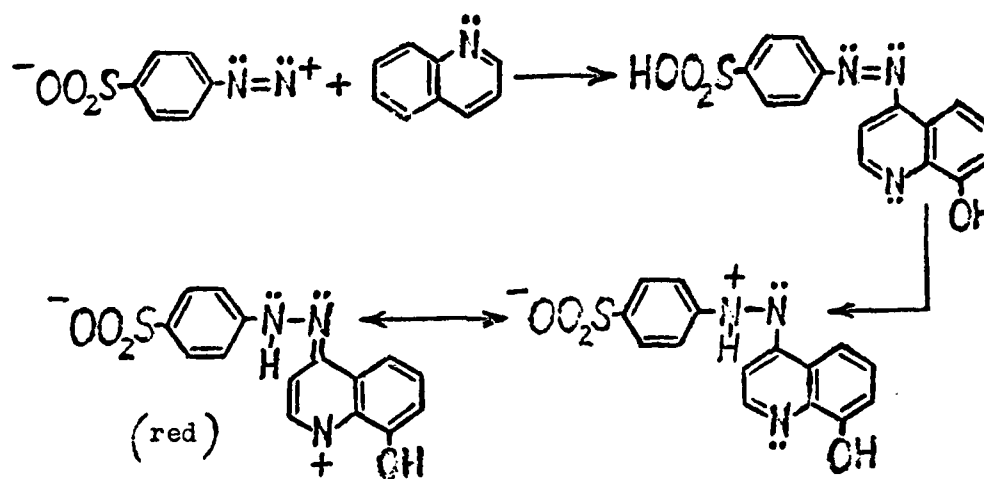
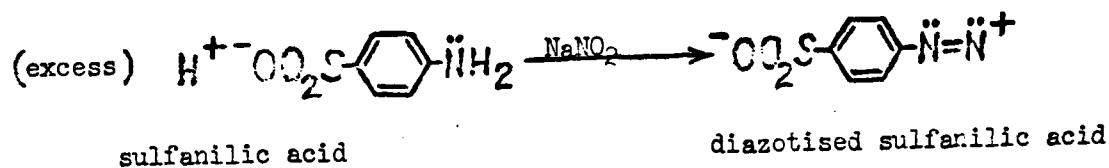
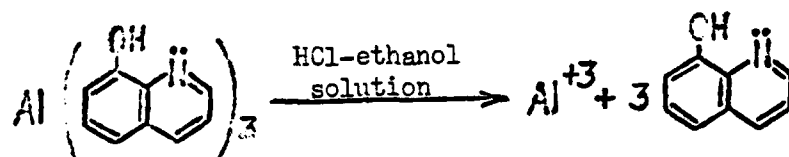
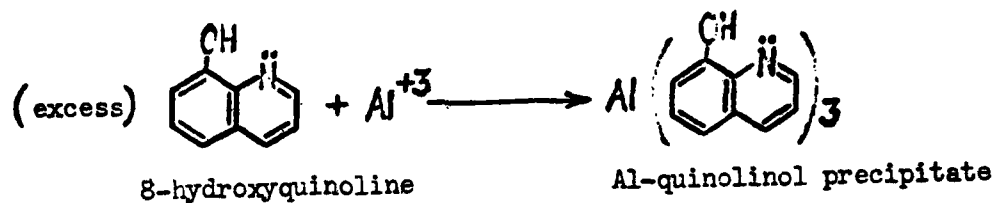
Solvent: Equal volumes of 2N HCl and 95% ethanol solutions.

Sulfanilic acid solution: 9.6 g. sulfanilic acid is dissolved in 1000 ml of warm 30% acetic acid.

Sodium nitrite solution: 2.85 g. sodium nitrite are dissolved in 1000 ml water.

Sodium hydroxide solution: 80 g sodium hydroxide are dissolved in 950 ml water.

REACTIONS INVOLVED



PROCEDURE

Each sea water sample was filtered through Whatman 4.25 cm GF/C medium-fine glass-fiber filter paper to remove particulate matter. A ten ml aliquot of each sample was then pipetted into a clean 25 ml flask. Each sample was made slightly acidic by adding 2 drops of 1:4 acetic acid: water solution and then buffered by adding 0.6 ml of sodium acetate solution. An excess of 8-hydroxyquinoline solution (0.3 ml) was added to precipitate all available aluminum, and each sample was then mixed by inverting several times and allowed to stand overnight.

The prepared samples were then heated in a water bath for 30 minutes each at 70°C and suction-filtered through a 100 millimicron Millipore filter (this small pore size was found to be necessary due to the extremely small size of the Al-quinolinol particles). The flask was rinsed with several small portions of boiling water and the washings filtered also.

The filter funnel containing the Al-quinolinol precipitate was then transferred to a 100 ml filter flask and 2 ml of hot HCl-ethanol solution was added to dissolve the precipitate. This was then suction-filtered and followed successively by washings of 2 small portions of hot distilled water, a 1.5 ml portion of HCl-ethanol solution, and 2 more portions of hot distilled water.

The solution in the flask was then transferred to a 50 ml volumetric flask. The filter flask was rinsed with 2 small portions of distilled water and the washings added to the volumetric flask. 1 ml of sulfanilic acid solution was added and this was diazotised with 1 ml of sodium nitrite solution. The flask was allowed to stand for 10 minutes, and 10 ml of NaOH solution was then added. A deep reddish color immediately developed, and when the solution was diluted by an appropriate factor (1/20 and 1/50 dilutions were used) the percent transmittance was easily resolvable on a Bausch & Lomb Spectronic 20 spectrophotometer set at a wavelength of 490 microns. Readings were compared to a blank which consisted of 3.5 ml HCl-ethanol solution, 1 ml sulfanilic acid solution, a ml sodium nitrite solution, 10 ml NaOH solution, and sufficient distilled water to fill a 50 ml volumetric flask.

A standard curve was prepared by measuring the absorbance of .10, .25, .50, .75, 1.00 and 1.50 ppm Al standard solutions and plotting the results (Figure 1). The aluminum concentration of each sample was then determined by comparing the absorbance values with those plotted on the standard curve (Table 1).

B. ATOMIC ABSORPTION DETERMINATION #1

To check the aluminum concentration values obtained by the colorimetric determination, portions of the same samples were analyzed for aluminum content using a Perkin-Elmer model 303 Atomic Absorption Spectrophotometer. However, due to the insensitivity of the instrument to aluminum concentrations as low as those present in sea water (the sensitivity for aluminum is 0.9 ppm for 1 percent absorption) it was first necessary to concentrate the aluminum in each sample by a factor of 100. This was accomplished by extracting the Al^{+3} from the sample with a solution of 8-hydroxyquinoline, diphenylthiocarbazone, and acetylacetone (2,4-pentanedione) dissolved in benzene, following the method described by Hsu and Pipes.⁵ Although their work was limited to Al^{+3} residues in distilled water solutions, the method was applied to seawater samples due to the limited volume of each sample available (about 750 ml) and the lack of an alternative method of accurately concentrating the aluminum present in the samples by such a large factor.

REAGENTS

Benzene solution: 0.75 g 8-hydroxyquinoline, 0.10 g diphenylthiocarbazone, and 20.0 ml acetylacetone were dissolved in benzene to a final volume of 100 ml in a volumetric flask.

PROCEDURE

500 ml of each sample (previously filtered through Whatman 4.25 cm GF/C filter paper) was pipetted into a 500 ml separatory funnel. The pH was adjusted to 4.8 using solutions of 1:4 acetic acid: water and saturated sodium acetate as required, and checked with a pH meter. Five ml of benzene-extraction solution was then pipetted in, and the funnel was stoppered, shaken vigorously for 1 minute, and then allowed to stand for 4 hours. Next, the aqueous phase was removed and the organic phase placed in a small ground-glass stoppered flask.

Standards were prepared by diluting a commercially available standard solution⁶ of 1000 ppm Al^{+3} (with $AlCl_3$ as substrate) to concentrations 20X less than those desired and then extracting 100 ml of each with 5 ml of benzene solution to obtain the desired concentrations. Standards of 10, 20, 50, 75, 100, and 150 ppm Al^{+3} in benzene solution were prepared in this way. Although this meant a difference of a factor of 5 in the extraction ratios of the standards versus samples (20:1 vs. 100:1) it was decided that this should have a near-negligible effect on the results due to the very low solubility of benzene in water (0.08 ml/100 ml).

All atomic absorption analyses were performed using a Perkin-Elmer model 303 Atomic Absorption Spectrophotometer equipped with an Al-Cu-Fe-Ti cathode ray lamp and a nitrous oxide-acetylene burner head. The wavelength was set at 3092.8A, and the flame height, fuel and oxidant flow, and rate of aspiration were adjusted to yield maximum sensitivity. The instrument was zeroed using the benzene solution as blank; all standards and samples were then aspirated and the absorption of each was recorded (Table 2). A standard curve was then prepared (Figure 2) and the sample values plotted to determine the concentration of each.

C. ATOMIC ABSORPTION DETERMINATION # 2

To further check the reliability of the atomic absorption method, the analysis was repeated, with some modifications, on 8 surface water samples (4 from the western Atlantic and 4 from the eastern Pacific) obtained during the summer of 1968 and stored under refrigeration in 1-liter polyethylene bottles. Two sets of standards were prepared; one set was extracted from dilutions prepared in distilled water so as to yield benzene solution standards of 10, 20, 50, and 75 ppm, and a second set was extracted from dilutions prepared in artificial sea water so as to yield benzene solution standards of 10, 20, 50, and 75 ppm. This was done to check the effect of salt water versus distilled water on the extraction process. The other notable modifications in this determination were that the pH of all aqueous samples and standards were unaltered, and that the extraction ratios of all samples and standards were 90:1 to preclude the possibility of benzene-water solubility effects.

REAGENTS

Benzene solution: 0.75 g. 8-hydroxyquinoline, 0.10 g. diphenylthiocarbazone, and 20.0 ml acetylacetone were dissolved in benzene to a final volume of 100 ml in a volumetric flask.

Artificial sea water: per 100 ml weigh out

2.6518g NaCl, 0.6767g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$,

0.5255 g $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, 0.1511g $\text{CaCl}_2 \cdot \text{H}_2\text{O}$,

0.0725g KCl, and 0.020g NaHCO_3

PROCEDURE

Of the eight samples analyzed, four were first filtered through a 47 mm diameter, .45 micron Millipore filter, two unfiltered samples were warmed for a short time, and to two other unfiltered samples 1 ml of glacial acetic acid was added prior to extraction. 900 ml of each sample was placed in a clean, dry 1000 ml round-bottomed flask and 10 ml of benzene solution was added. The flask was stoppered, shaken vigorously for 1 minute and then allowed to stand for 1 hour, at which time it was shaken again for 1 minute and allowed to stand overnight. The aqueous phase was then removed and the organic phase placed in a glass vial and tightly sealed.

Benzene solution standards were prepared by diluting 1000 ppm Al^{+3} standard solutions prepared in distilled water (with $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ as substrate) to 10 ppm and then adding aliquots of this to distilled water in a 1000 ml round-bottomed flask to produce a 900 ml solution of 1/90 the desired concentration of the final benzene standard. These solutions were then extracted with 10 ml of benzene solution in the same manner as the seawater samples. This produced standards of 10, 20, 50, and 75 ppm Al^{+3} which were stored in tightly capped glass vials.

This procedure was repeated with a 1000 ppm Al^{+3} solution prepared in artificial seawater and diluted with prepared artificial seawater to produce 900 ml volumes of the desired Al^{+3} concentration. These standards were then extracted in the same manner and also stored in vials.

All samples and standards were then analyzed on the same instrument as the previous determination, again with all controls adjusted for maximum sensitivity. The absorptions of each were recorded and standard curves prepared (Figure 3 and 4) in the same manner as the previous determination. Results are contained in Table 3.

RESULTS

Results of the colorimetric determination showed the concentration of aluminum in the water column to generally increase with depth. Values ranged from a low of 0.2 ppm at the surface to about 0.4 ppm at depths over 2000 meters. The only exception to this trend was the sample obtained at 1500 m, which showed a concentration of only about 0.1 ppm; the reason for this discrepancy is unknown. Some small variations did occur in values obtained from the 1/20 dilution versus those obtained from the 1/50 dilutions, probably due to slight mechanical error in the dilution process since percent transmittance was very sensitive to slight changes in concentration in this range.

The first atomic absorption analysis, however, yielded values which were significantly lower than those obtained by the colorimetric method. Concentrations ranged from a low of .089 ppm Al at the surface to .122 ppm at 2500 m, with the exception of a high reading of .123 ppm at 800 m, and, as with the colorimetric determination, a low value (.100 ppm) at 1500 m. Again, the results indicate a general increase in the aluminum concentration with depth, although the concentration values measured do approach the sensitivity limitations of the instrument. A plot of depth versus concentration for both determinations is shown in Figure 5.

Results of the second atomic absorption analysis were disappointing. Absorption readings for all eight of the 4 year old surface samples, filtered and unfiltered, were virtually indiscernible from the blank. However, readings for both the distilled water and artificial seawater standards were very similar to those obtained for the distilled water standards prepared for the first atomic absorption analysis, which means that the sensitivity of the instrument was about the same for both analyses. Thus, the great disparity between the results of this analysis and those performed on the first set of seawater samples lies in the nature of the samples, not in the performance of the instrument. The sensitivity of the instrument, in fact, was excellent for all of the atomic absorption analyses.

CONCLUSIONS

Although the difference between the Al concentration values arrived at by the colorimetric and atomic absorption analyses was great enough to cast some doubt on the precise concentration of each Ocean Acre sample, the combined results are close enough to limit such concentrations to a very narrow range. Of course, as previously mentioned, it is very probable that localized variations in the concentration of aluminum in seawater do occur, but the fact that this set of samples was obtained far from the continental shelf should make it fairly representative of most areas of the open ocean.

As for the low results obtained in the second atomic absorption analysis, it can only be concluded that this was due to the nature of the samples and not to any failure on the part of the analytical method, since the sensitivity of the instrument was very good and readings for both sets of standards were very similar to those obtained for the standards prepared for the first analysis. The poor agreement may be due in part to the fact that all 8 samples analyzed were surface samples, where the aluminum concentration appears to be lowest, and that all 8 samples had been in storage for four years and, despite refrigeration, the loss of Al^{+3} due to adsorption on the walls of the container and precipitation is probable.

Still, the combined results of all three analyses do yield some important conclusions. First, the trend of increasing aluminum concentration with increasing depth is borne out by the results of both the colorimetric and atomic absorption determinations, and agrees with the findings of Haendler and Thompson.

Secondly, the close correlation between the absorption readings of the atomic absorption standards derived from both distilled water and artificial seawater as well as the excellent sensitivity achieved with all three sets of standards, indicate that the benzene extraction method is a very feasible and practical means of accurately concentrating the available aluminum in seawater, as well as in fresh or distilled water. This procedure brings the aluminum concentration in such samples within the detection limits (greater than .9 ppm for 1 percent absorption) of the atomic absorption spectrophotometer.

The fact that slightly higher, but similar, results were obtained by the colorimetric analysis does not belie the use of the colorimetric method. Although the reason for the discrepancy in the concentration values obtained by the two analyses is not known, the colorimetric method appears to be an accurate method for the direct analysis of water samples containing extremely low concentrations of aluminum (as low as 0.001 ppm Al, versus a limit of 0.01 ppm Al for the atomic absorption method).

To check these results, we would recommend further study utilizing both of these methods. Samples should be as fresh as possible and at least 2 liters in volume to produce a final benzene solution sample of adequate volume for atomic absorption analyses. I would also recommend that a higher benzene solution/seawater extraction ratio be used (perhaps 1:200) to insure that the absorption readings of all samples fall well within the detection limits of the instrument; Hsu and Pipes⁵ claim that extraction ratios up to 1:1000 are feasible, and that the limit is based more on the availability of special separation apparatus rather than on the method itself. Samples, if possible, should be obtained from known depths at various locations in the open ocean where discrepancies in results caused by suspended particulate matter and localized concentrations effects would be minimal. This hopefully, would provide more uniform results as well as provide a further check of the consistency of the two methods of analysis.

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TABLE 1

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ALUMINUM CONCENTRATION OF SEAWATER

OCEAN ACRE SAMPLES										
STANDARD	PERCENT TRANSMITTANCE				AVERAGE		ABSORBANCE		CONCENTRATION	
(Run)	1		2						(ppm)	
(dilution)	1/20	1/50	1/20	1/50	1/20	1/50	1/20	1/50		
1	54.8	80.6	58.8	83.3	56.8	81.9	.2534	.0867	0.10	
2	48.5	75.8	48.5	77.1	48.5	76.4	.3143	.1169	0.25	
3	39.8	69.9	41.3	72.1	40.5	71.0	.3925	.1487	0.50	
4*	44.5	73.1			44.5	73.1	.3516	.1361	0.75	
5*	41.2	71.8			41.2	71.8	.3851	.1439	1.00	
6*									1.50	

SAMPLES	PERCENT TRANSMITTANCE				AVERAGE		ABSORBANCE		ACTUAL CONCENTRATION	
(Run)	1		2						(ppm)	
(dilution)	1/20	1/50	1/20	1/50	1/20	1/50	1/20	1/50	1/20	1/50
100m	50.9	78.3			50.9	78.3	.2933	.1062	.192	.187
800m	47.3	74.2			47.3	74.2	.3251	.1296	.281	.340
1200m	46.0	74.1			46.0	74.1	.3372	.1302	.317	.345
1500m**	58.8	80.9	53.1	80.1	55.9	80.5	.2526	.0942	.098	.131
1800m	43.3	72.6			43.3	72.6	.3635	.1391	.402	.417
2500m	44.6	72.8			44.6	72.8	.3507	.1379	.360	.406

* Transmittance much higher than expected - reason unknown.

** High transmittance checked by second run - this was also high.

TABLE 2

ATOMIC ABSORPTION SPECTROSCOPY DETERMINATION
 OF ALUMINUM CONCENTRATION OF SEAWATER

OCEAN ACRE SAMPLES

(8/21/72)

STANDARD	ABSORPTION*		AVERAGE	ABSORBANCE	CONCENTRATION (ppm)	SENSITIVITY (EXP ≤ 0.9)
	1	2				
1	13.9	14.3	14.1	.0660	10.0	0.67
2	28.6	27.4	28.0	.1427	20.0	0.62
3	52.1	50.6	51.3	.3125	50.0	0.70
4	70.7	70.7	70.7	.5331	75.0	0.62
5	71.6	71.0	71.3	.5421	100.0	0.81
6	79.1	81.0	80.0	.6990	150.0	0.94

SAMPLE (depth, m)	ABSORPTION*		AVERAGE	ABSORBANCE	CONCENTRATION (ppm)	ACTUAL CONCENTRATION (ppm/100)
	1	2				
100	12.8	12.6	12.7	.0590	8.9	.089
800	17.4		17.4	.0830	12.3	.123
1200	15.7	15.4	15.5	.0731	10.9	.109
1500	13.8	14.6	14.2	.0665	10.0	.100
1800	16.8	14.6	15.7	.0742	11.0	.110
2500	17.1		17.1	.0814	12.2	.122

* Number of readings taken was few due to small volume of final extracted solutions available.

TABLE 3

ATOMIC ABSORPTION SPECTROSCOPY DETERMINATION
OF ALUMINUM CONCENTRATION OF SEAWATER

SURFACE SAMPLES

(10/13/72)

STANDARD	ABSORPTION			AVERAGE	ABSORBANCE	CONCENTRATION (ppm)	SENSITIVITY (EXP ≤ 0.9)
	1	2	3				
D-1 ¹	18.8	19.7	18.8	19.1	.0921	10.0	0.48
D-2 ¹	32.1	34.6	33.7	33.5	.1772	20.0	0.50
D-3 ¹	55.2	57.8	57.3	56.8	.3645	50.0	0.60
D-4 ¹	70.5	71.7	69.9	70.7	.5331	75.0	0.62
S-1 ²	17.5	18.8	17.6	18.0	.0862	10.0	0.51
S-2 ²	33.3	30.9	32.6	32.3	.1694	20.0	0.52
S-3 ²	62.9	61.7	61.8	62.1	.4214	50.0	0.52
S-4 ²	66.4	64.1	64.0	64.9	.4547	75.0	0.72
SAMPLE	ABSORPTION			AVERAGE	ABSORBANCE	CONCENTRATION (ppm)	ACTUAL CONCENTRATION (ppm/90)
	1	2	3				
S-1 ³	0.6	0.4	0.5	0.5	.0022	↑ BELOW DETECTION LIMITS OF ANALYSIS ↓	↑ ↓
S-2 ³	0.6	0.3	0.4	0.4	.0017		
S-3 ³	0.2	0.1	0.2	0.2	.0009		
S-4 ³	0.2	0.1	0.0	0.1	.0004		
S-5 ⁴	0.5	0.3	0.4	0.4	.0017		
S-6 ⁴	0.4	0.2	0.2	0.3	.0013		
S-7 ⁵	0.1	0.0	0.1	0.1	.0004		
S-8 ⁵	0.0	0.0	0.0	0.0	.0000		

- 1 Extracted from standards prepared in distilled water.
- 2 Extracted from standards prepared in artificial seawater.
- 3 Suction filtered through 47mm diameter, 0.45 mμ pore Millipore filter
- 4 Unfiltered, warmed
- 5 Unfiltered, 1 ml glacial acetic acid added to each sample.

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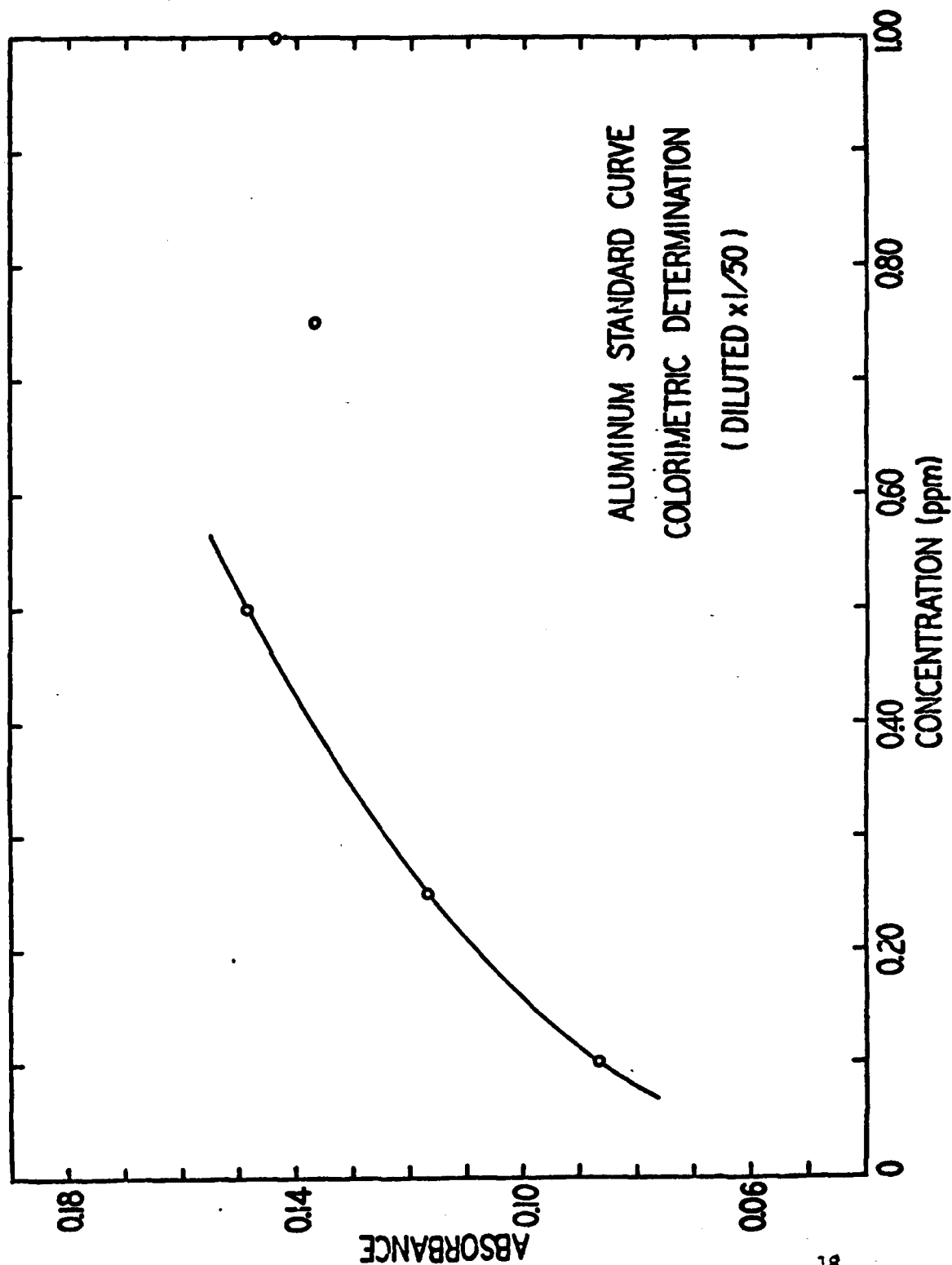


FIGURE 1a

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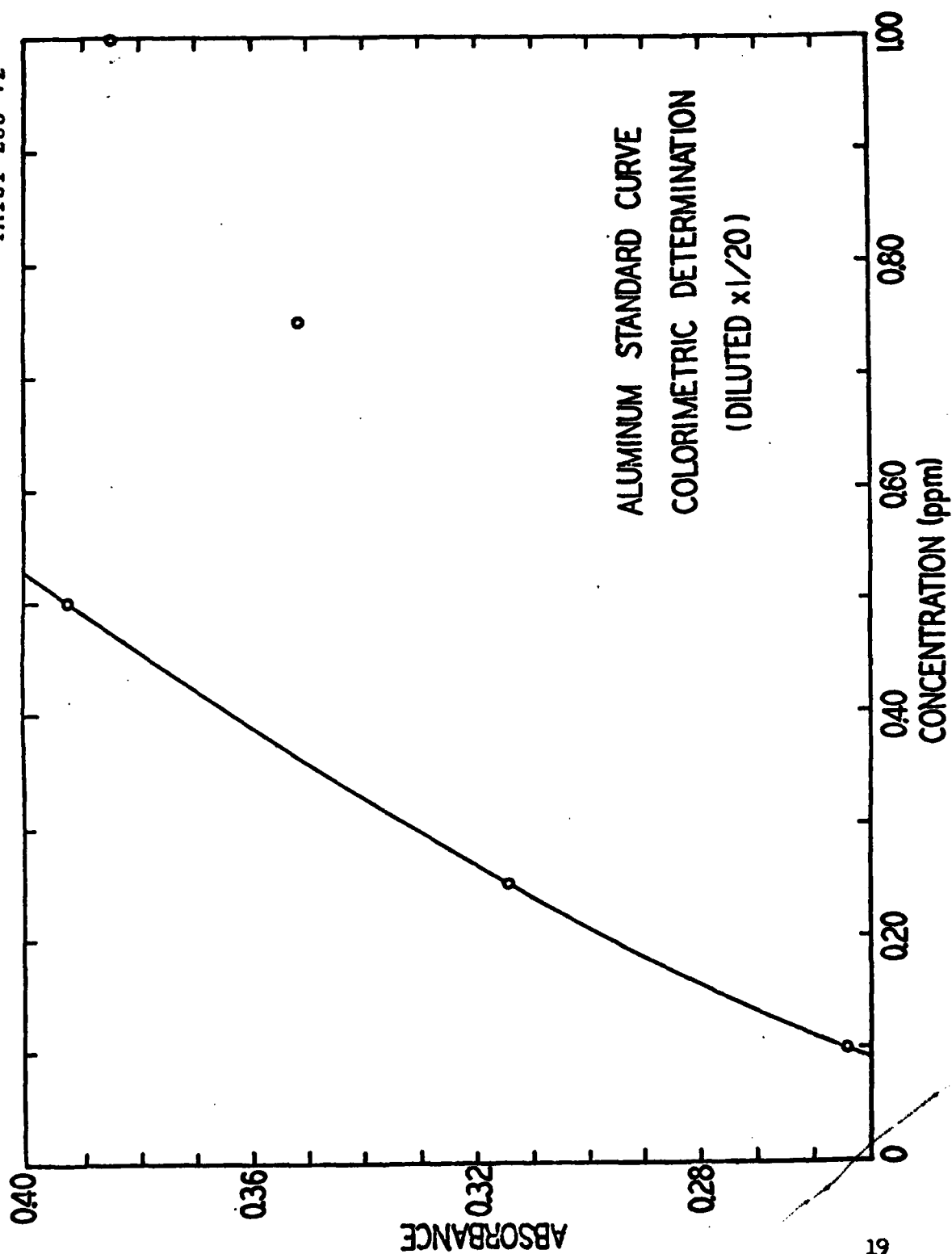


FIGURE 1b

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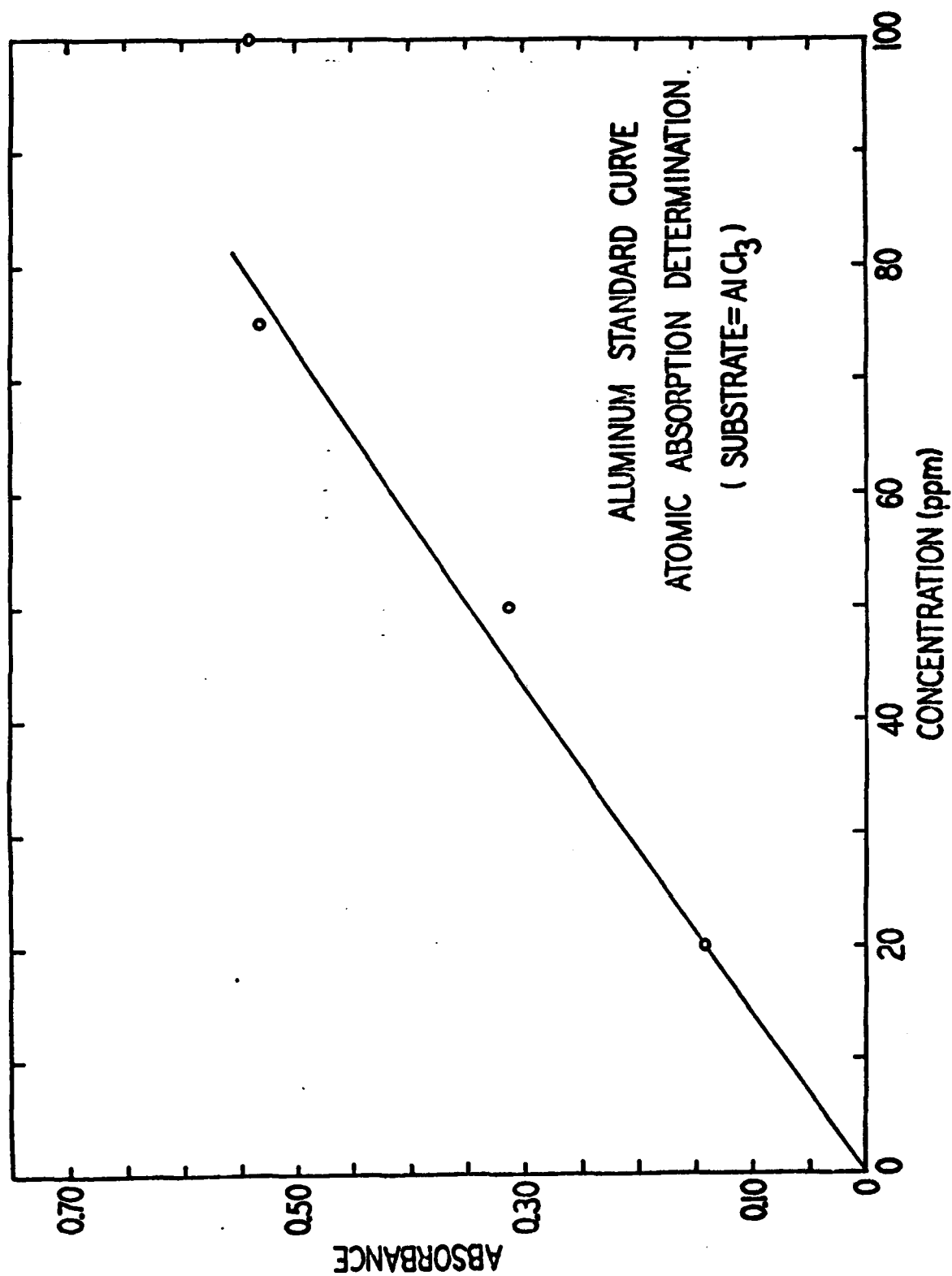


FIGURE 2

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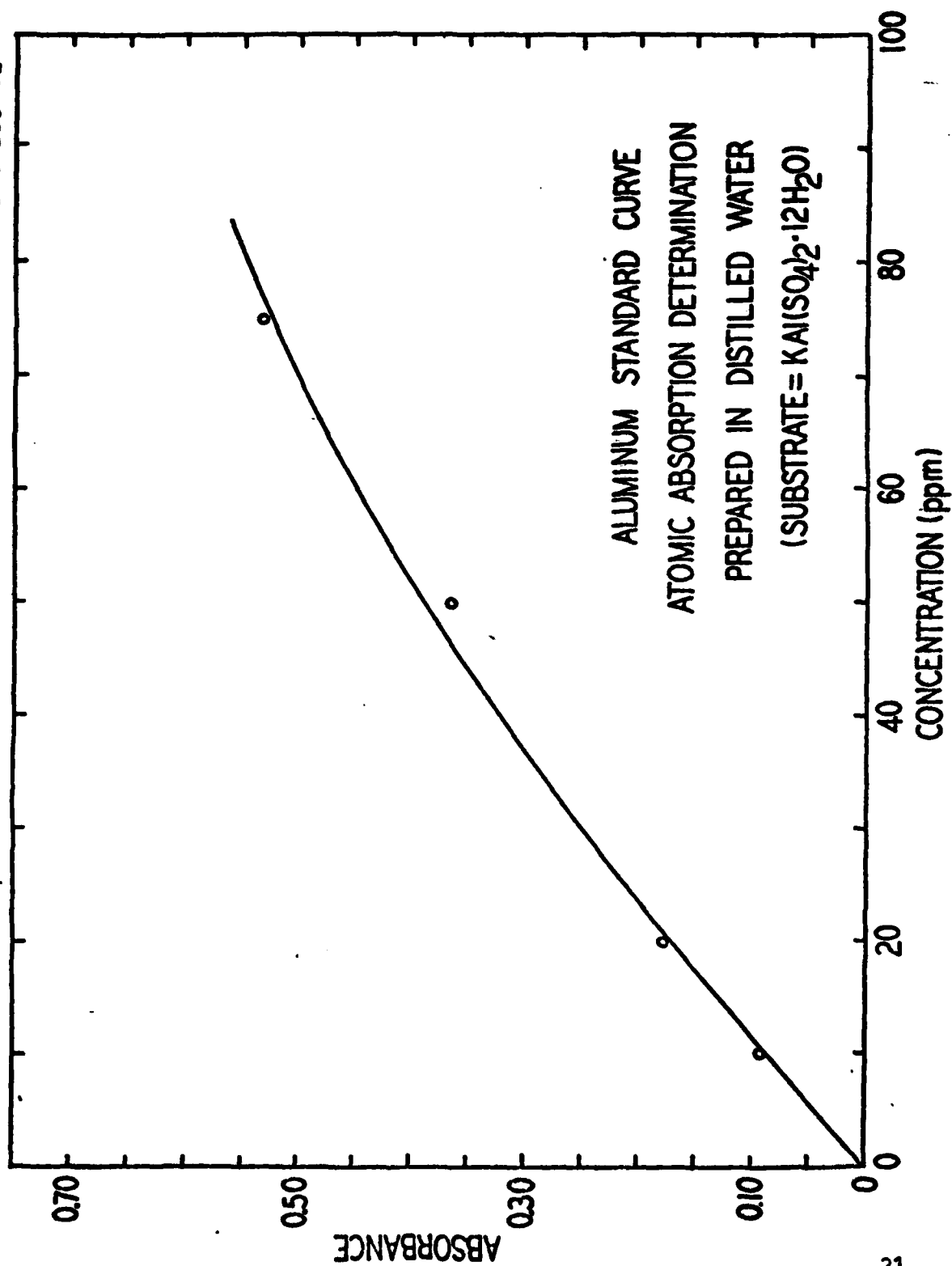


FIGURE 3

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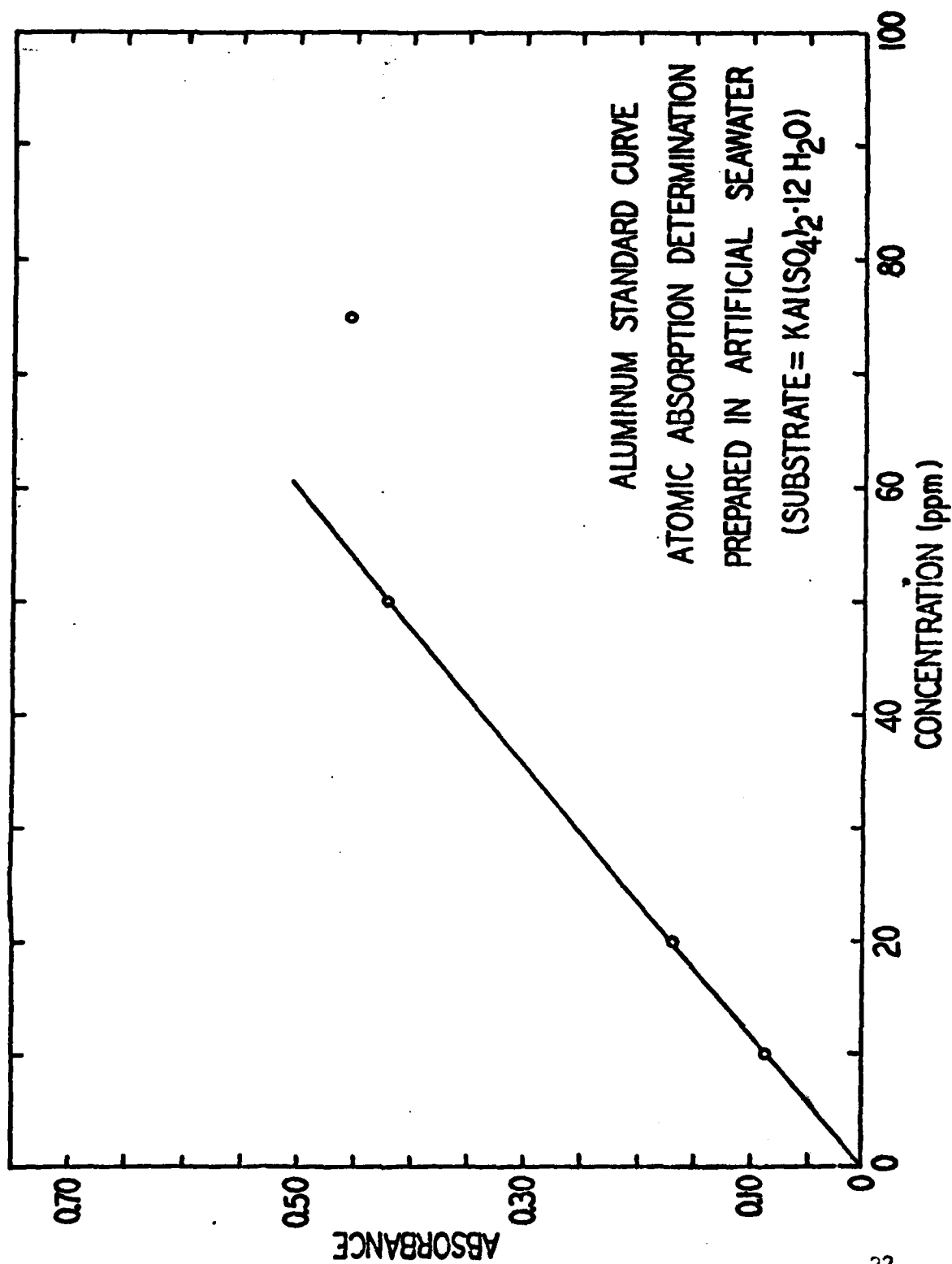


FIGURE 4

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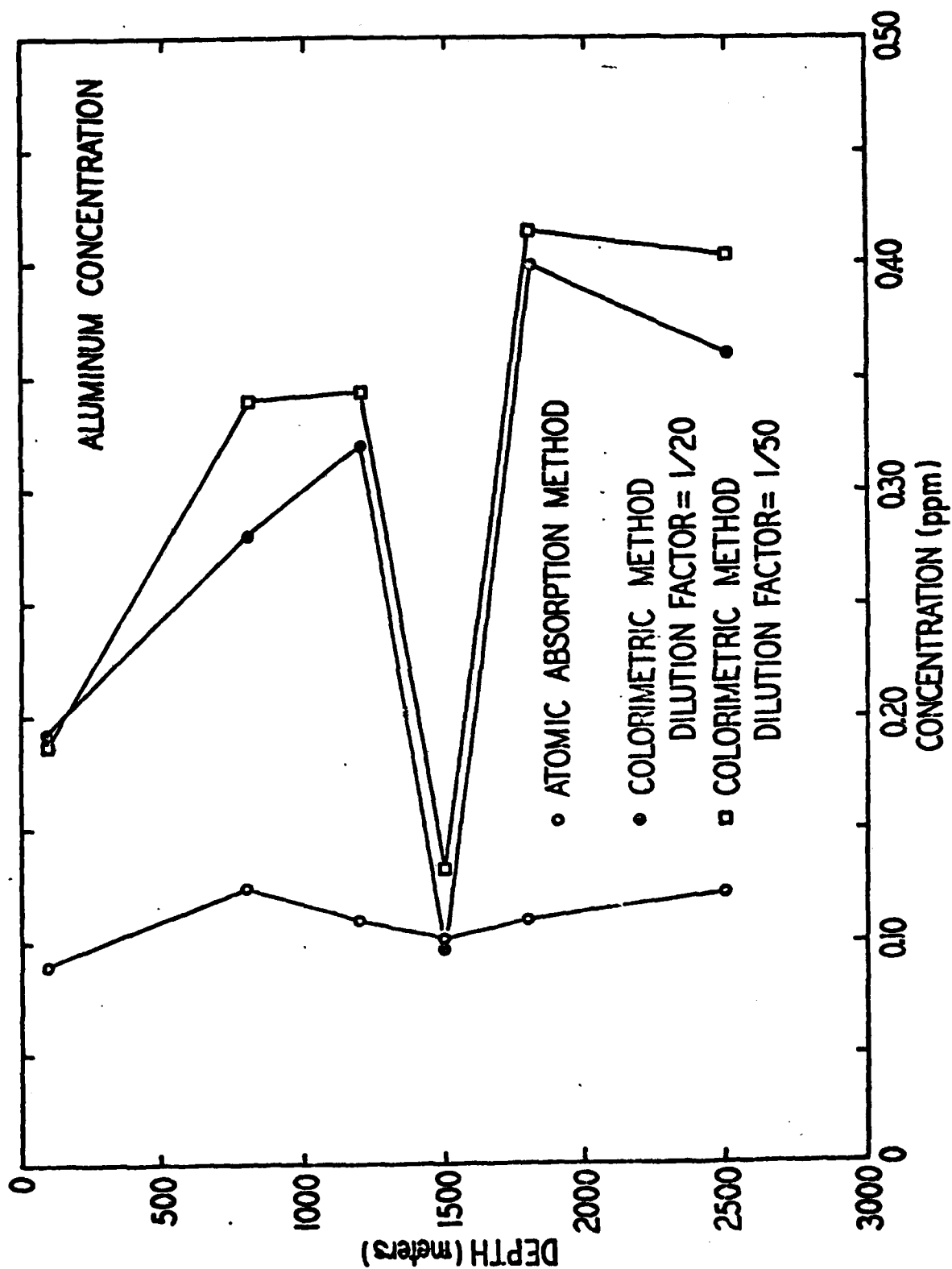


FIGURE 5

Determination of Aluminum Concentration in Seawater by Colorimetry
and Atomic Absorption Spectroscopy

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Ocean Science Department

TM No. TA131-285-72

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